## IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re application of Rakowski :

**OXIDATION RESISTANT FERRITIC** 

Group Art Unit 1742 :

STAINLESS STEELS

Serial No. 10/654,203

Confirmation No. 5809

Filed September 3, 2003

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Examiner Jessee Roe

## **DECLARATION OF MICHAEL P. BRADY**

Pittsburgh, Pennsylvania 15222-2312 August 23, 2007

Commissioner for Patents P.O. Box 1450 Alexandria, VA 22313-1450

Sir:

- 1. I, Michael P. Brady, declare as follows:
- 2. I am a citizen of the United States and currently reside at 110 Clemson Drive, Oak Ridge, TN 37830.
- 3. I am over the age of eighteen and am competent to make the statements in this Declaration.
- 4. I received a Ph.D. degree in Materials Science and Engineering from the University of Florida in August , 1993.
- 5. During 1993-1997, I was a National Research Council Postdoctoral Fellow at NASA's Glenn Research Center in the Environmental Durability Branch of the Materials Division and pursued research in high temperature alloy design and oxidation. In 1997, I joined the Materials Science and Technology Division at Oak Ridge National Laboratory as an ORISE Postdoctoral Fellow. I became a full time R&D staff member

at ORNL in 1998, and was promoted to Senior R&D staff in 2004. I am currently pursuing research in the development of corrosion-resistant, high temperature materials for cleaner and more efficient energy conversion and combustion systems, and the use of corrosion reactions to synthesize functional services for improved energy-related electrochemical systems and devices such as fuel cells. In fiscal year 2007, I am the lead/co-principle investigator for over \$3 million of research activities.

- 6. I am the author/coauthor of 5 issued U.S. Patents, 8 Pending U.S. Patents or ORNL Invention Disclosures, and over 80 publications in the areas of oxidation, alloy design, fuel cells, and coatings. Approximately half of my patents/invention disclosures and are in the area of stainless steels, with 3 specifically directed to fuel cell issues. I am the author/coauthor of more than 15 archival journal articles specifically in the areas of stainless steels or fuel cells, including a recent paper on alumina-forming stainless steels in the international multidisciplinary journal *Science*, as well as specialty publications in leading metallurgical journals such as *Oxidation of Metals, Scripta Materialia*, *Journal of Power Sources, Metallurgical and Materials Transactions*, and *International Journal of Hydrogen Energy*. I also serve on the international advisory board of the journal Oxidation of Metals, and act as a technical reviewer for virtually every major metallurgical related journal.
- 7. I have specifically led efforts devoted to the evaluation and development of ferritic stainless steels (and related alloys) for SOFC fuel cells. I have conducted oxidation studies of ferritic stainless steels, as well as austenitic, Ni-base, Cr-base, and intermetallic alloys. I am well versed in surface preparation methods, including mechanical abrasion and electropolishing.
- 8. I have thoroughly reviewed U.S. Patent Application Serial No. 10/654,203 to Rakowski (the "Rakowski application"). The Rakowski application describes a method of electrochemically modifying a surface of a ferritic stainless steel to improve the oxidation resistance of the surface when exposed to high temperature, oxidizing environments, such as the environment within a solid oxide fuel cell ("SOFC"). Electropolishing is a well known method of electrochemically modifying a steel surface and operates by removing material from the surface to flatten (smooth) the surface. A

benefit of electropolishing is that the smoothed surface better reflects light and, therefore, the appearance of the surface is improved. It can also be used to minimize surface defects to improve other properties such as fatigue resistance. I am not aware of any previous findings of electropolishing enhancing high temperature oxidation resistance.

- 9. At a time just prior to September 3, 2003, metallurgists conventionally believed that the high temperature oxidation resistance of a ferritic stainless steel surface, as well as other classes of high temperature alloys, would not be improved by electropolishing the surface. Instead, metallurgists conventionally believed that mechanically deforming (roughening) the surface of a stainless steel would introduce cold work and dislocations, resulting in a fine local surface grain size on heating to high temperatures, that would generally improve oxidation resistance by increasing outward diffusion of the protective scale forming element (typically Cr or Al). It was believed that highly polished surfaces, either by mechanical polishing or electropolishing, result in a low defect surface microstructure, which does not recrystallize to a fine local grain size and therefore does not provide preferential outward diffusion paths to enhance protective oxide scale formation. It is general practice in the oxidation research community to perform oxidation tests with mechanically abraded surfaces, typically in the 600 grit surface finish range. The conventional beliefs regarding surface roughness, grain size refinement, and oxidation resistance in alloys have been prevalent for decades, for example refer to the references C. S. Giggins et al., "The Effect of Alloy Grain-Size and Surface Deformation on the Selective Oxidation of Chromium in Ni-Cr Allovs at Temperatures of 900°C and 1000°C", 245 Transactions of the Metallurgical Society of AIME at 2509-2514 (December 1969); and J. M. Rakowski et al., "The Effect of Surface Preparation on the Oxidation Behavior of Gamma TiAl-Base Intermetallic Alloys", 35 Scripta Materialia at 1417-1422 (1996). Both of these references suggest the advantage of a mechanically deformed surface in regards to establishing the desired continuous, protective oxide surface scale (chromia or alumina) to enhance oxidation resistance.
- 10. In light of the conventional beliefs regarding electropolishing discussed in paragraph 9 above, I was very surprised by the unexpected oxidation test results

described in the Rakowski application. The Rakowski application describes testing wherein the oxidation resistance of certain ferritic stainless steels was significantly improved by electropolishing surfaces of the steel, and also was improved relative to samples of the steel processed to have relatively rough surfaces. I believe that the results described in Rakowski were entirely unexpected prior to September 3, 2003, and were directly opposite to what a metallurgist would have predicted at that time based on the conventional beliefs in the field, described in paragraph 9 above. At a time just prior to September 3, 2003, metallurgists would have predicted that ferritic stainless steel surfaces that were electropolished would exhibit no beneficial oxidation-related effects, and likely a reduced level of oxidation resistance.

- 11. I believe that the results described in the Rakowski application are significant in that the inventor discovered that electropolishing provides a ready technique for further enhancing the oxidation resistance of surfaces of certain ferritic stainless steels.
- 12. I further declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements are made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or document or any registration resulting therefrom.

Date:	8/23/07	Michael P Brady

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